341

N64-30607

Code None
Cat-18

Investigation of Room-Temperature Slip in Zone-Melted Tungsten Single Crystals

R. G. Garlick and H. B. Probst

6p. 30607 ABST

Tungsten single-crystal specimens of various orientations were deformed in tension at room temperature. Slip traces indicated both {112}⟨111⟩ and {110}⟨111⟩ slip; however, about 10 pct plastic deformation was required before these traces could be seen. Resolved shear-stress considerations indicated that at least some of the particular systems observed were not those on which slip initiated but were secondary systems. This is highly probable, considering the high plastic strains involved. Analysis of the stress data with respect to possible slip systems indicated that slip must have been initiated at stresses below the proportional-limit stresses measured.

Author

ALTHOUGH there is general agreement that slip deformation in fcc and hcp metals takes place on the plane of close packing and in the direction of close packing, there is no such agreement for slip in bcc metals. It is generally agreed that the close-packed direction ⟨111⟩ is the slip direction, but investigations have led to conflicting descriptions of the slip plane. As Hoke and Maddin[1] point out, there are essentially four points of view.

1) Deformation in bcc metals is composite shear on {112} and {110} planes or on nonparallel {110} planes, and any apparent slip on other planes can be accounted for completely by slip on these planes. Chen and Maddin have reported this to be the possible case for molybdenum.[2]

2) Slip occurs in the ⟨111⟩ direction and is limited to {110}, {112}, and {123} planes (the three closest packed planes of the lattice).

3) Slip occurs in a ⟨111⟩ direction, but not necessarily on planes of close packing (banal slip).

4) Slip occurs in those directions and on those planes for which β, the ratio of Burgers vector to interplanar spacing, is a minimum.

The slip systems of the bcc metal tungsten have never been fully determined, although several investigators have reported pertinent results.[3-7]

The present work was initiated to determine the active slip systems in tungsten at room temperature, the critical resolved shear stresses associated with these systems, and the orientation dependency of operative systems. These features of tungsten deformation at room temperature have never been reported in the literature. The authors find this somewhat surprising, particularly in view of the availability of tungsten single crystals since the advent of electron-beam zone-melting techniques and the widespread interest in refractory metals. It would seem apparent that such work has been attempted but the results have not been reported, possibly due to the conflict with deformation theory. The results of the present work conflict with any straightforward description of deformation based on the proposed explanations of slip in bcc metals. Although these conflicts have not been resolved, a presentation of the problem seems in order and should be of value to others working in this field.

R. G. GARLICK and H. B. PROBST are Research Metallurgist, Refractory Metals Section, and Head, Refractory Compounds Section, respectively, Lewis Research Center, National Aeronautics and Space Administration, Cleveland, Ohio.

Manuscript submitted January 31, 1963. IMD

MATERIALS AND PROCEDURE

Single-crystal tungsten rods of 1/8-in. diameter were grown from undoped commercial rod by one-pass electron-beam zone melting in apparatus previously described by Witzke.[8] Representative analyses of the rod before and after melting are given in Table I. There was no detectable zoning of impurities during melting.

Buttonhead tensile specimens, as shown in Fig. 1, were machined from single crystals. Two flats 90 deg apart were ground in the gage length of each specimen. These flat surfaces facilitated the analysis of slip traces. No attempt was made to position these flats with respect to the crystal orientation. The worked surface layer of the machined specimens was removed by electrolytic polishing in a NaOH solution. Tensile-axis orientations were determined by standard Laue back-reflection techniques. The sharpness of the Laue spots indicated that the crystals were free of strain.

Table I. Typical Impurities, Ppm

Element	Starting Material	Zone-Melted
C	10	10
O	4	2
N	10	8
Al	2.5 ± 1.0	0.1 ± 0.1
Cr	0.3 ± 0.15	0.55 ± 0.2
Cu	0.13 ± 0.1	0.15 ± 0.1
Fe	5.0 ± 1.0	0.7 ± 0.2
Mo	104 ± 10	44 ± 20
Ni	1.2 ± 0.2	0.35 ± 0.15

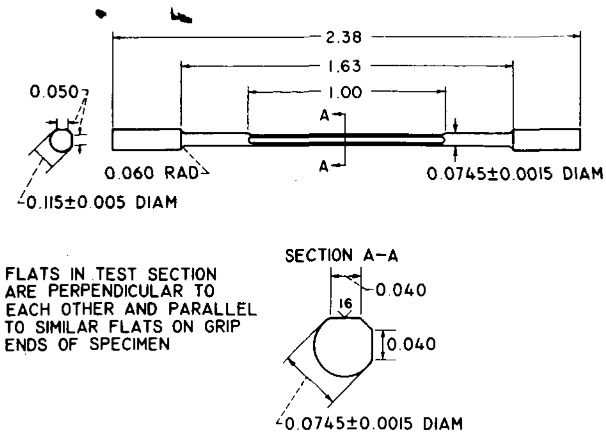

Fig. 1—Buttonhead tensile specimen (all dimensions in inches).

Specimens were deformed in tension at a strain rate of 0.01 in. per in. per min. Since no actual yield-point drop was observed, the proportional-limit stress was taken as the criterion for the onset of plastic flow. Appreciable plastic deformation (about 10 pct uniform elongation, which corresponded to a stress near the ultimate) was necessary before slip lines could be observed with an optical microscope. Slip traces were indexed by using the two-trace method described by Barrett.[9]

Several specimens were deformed in tension at 2500°F (~1650°K). These tests were performed in a vacuum of 5×10^{-5} mm Hg at a strain rate of 0.01 in. per in. per min. The specimens were not pulled to fracture, deformation being stopped after about 20 pct elongation. The tensile-axis rotation during testing was determined by analysis of back-reflection Laue patterns taken before and after deformation.

RESULTS

Crystals of those tensile-axis orientations shown in Fig. 2 were studied. Analysis of slip traces separated the crystals into two groups according to apparent slip-plane orientations. Fig. 3 is a plot of the poles of the slip planes as determined from the predominant traces of the specimen flats. Crystals with orientations near the $(011)(\bar{1}11)$ tie line (J, K, L, N, O, P, hereinafter referred to as Group 1) exhibited predominant slip-lane traces near $(\bar{1}10)$ with the exception of J, which showed an apparent slip plane near (011). The slip traces of Group 1 crystals were straight and generally well-defined. Orientations nearer (001) (A through I, hereinafter referred to as Group 2) gave visible slip traces near $(1\bar{1}2)$. These traces were wavy and not easily resolved. The crystal of tensile-axis orientation near (011) (crystal M) showed slip-trace markings near $(\bar{1}01)$ and shall be discussed separately, since stress data warrant classifying this crystal differently from Groups 1 and 2. In all cases, there is no doubt that the observed markings were slip lines. Prior to their appearance the sample surfaces were completely featureless. Also, the markings could be removed by electropolishing and made to reappear by subsequent straining.

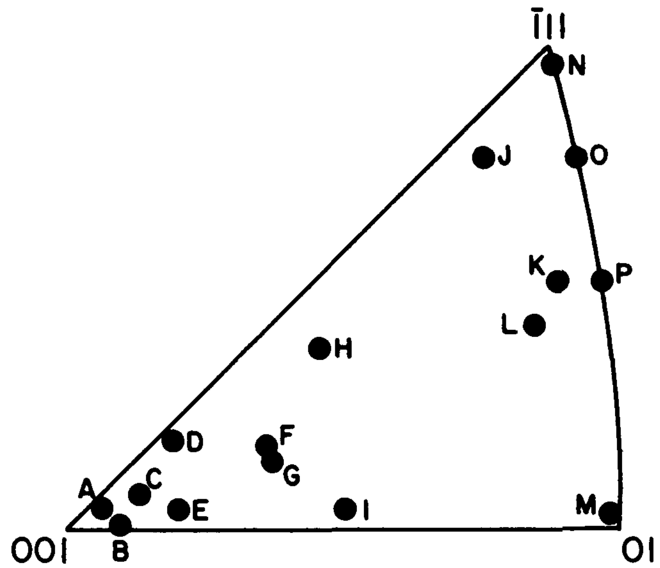

Fig. 2—Crystal orientations.

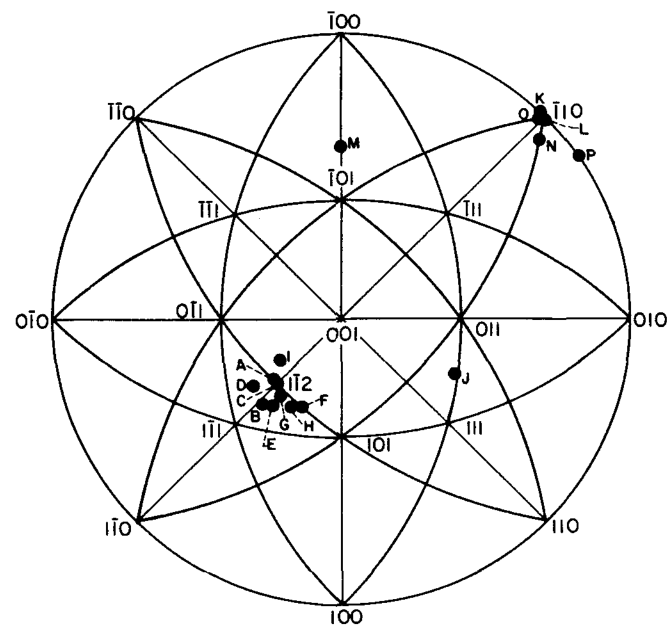

Fig. 3—Slip planes as determined from predominant traces.

Many specimens showed evidence of duplex slip. When two sets of slip lines were observed on a specimen flat, the most predominant set of traces was assumed to be those of the plane on which the major deformation had occurred, and the planes plotted in Fig. 3 represent these traces. When it was possible to index two sets of slip traces, they were usually from conjugate slip systems.

Tensile data from all specimens are summarized in Table II. Fig. 4 shows the stress-strain curve for specimen M and stress-strain curves for specimens typical of Groups 1 and 2. The strain indicated is actually the crosshead displacement of the Instron Testing Machine. Specimen M showed a

Table II. Tensile-Test Data

	Specimen	Proportional-Limit Stress, Lb Per Sq In.	Slip Plane Indicated by Trace	Angle from Tensile Axis to Slip Plane	Slip Direction	Angle from Tensile Axis to Slip Direction	Resolved Shear Stress, Lb Per Sq In.
Group 1	J	63,400	011	29	$\bar{1}\bar{1}1$	66	22,500
	K	65,700	$\bar{1}10$	50	111	55	24,200
	L	74,500	$\bar{1}10$	51.5	111	52	28,500
	N	87,000	$\bar{1}10$	39	111	66	27,300
	O	55,200	$\bar{1}10$	39.5	111	59	22,500
	P	68,600	$\bar{1}10$	45	111	54	28,200
							Av. 25,500
Group 2	A	27,250	$1\bar{1}2$	38	$\bar{1}11$	52	13,250
	B	28,500	$1\bar{1}2$	38	$\bar{1}11$	52	13,800
	C	32,800	$1\bar{1}2$	44	$\bar{1}11$	49	15,400
	D	26,850	$1\bar{1}2$	46.5	$\bar{1}11$	43.5	13,400
	E	30,850	$1\bar{1}2$	45.5	$\bar{1}11$	44.5	15,400
	F	26,500	$1\bar{1}2$	51.5	$\bar{1}11$	39.5	12,700
	G	28,250	$1\bar{1}2$	53	$\bar{1}11$	38	13,400
	H	31,600	$1\bar{1}2$	61	$\bar{1}11$	30	13,250
	I	24,900	$1\bar{1}2$	52	$\bar{1}11$	40	11,700
							Av. 13,600
	M	132,000	$\bar{1}01$	59	111	36	55,000

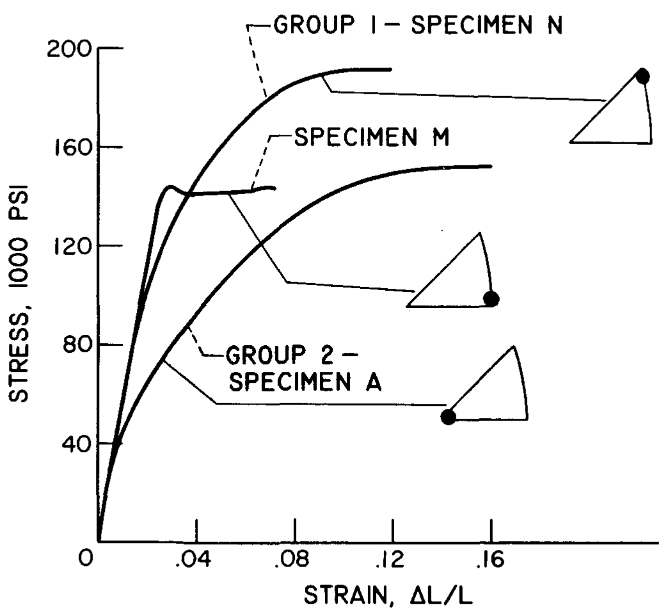

Fig. 4—Representative tensile curves.

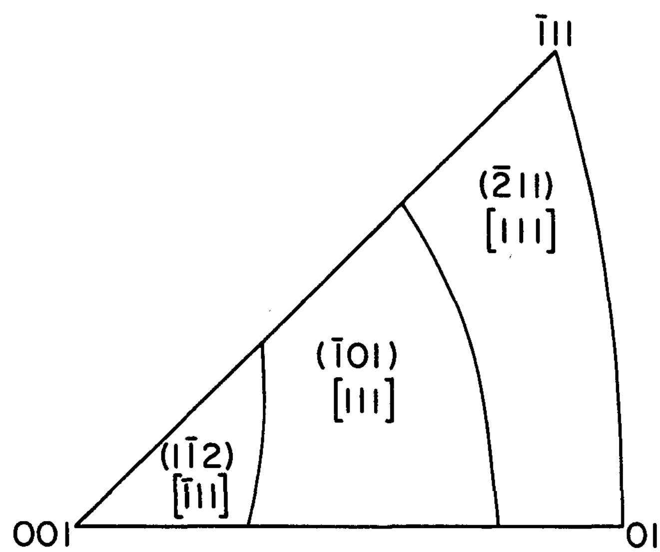

Fig. 5—Prevailing slip systems with equal critical resolved shear stresses for $\{112\}\langle 111\rangle$ and $\{110\}\langle 111\rangle$ slip systems assumed.

definite yield point and exhibited no strain hardening while neither Group 1 nor Group 2 crystals showed any yield point. Crystals of both Group 1 and Group 2 exhibited strain hardening, with Group 1 crystals showing a higher strain-hardening rate. The yield stress for specimen M was much higher than the proportional-limit stresses for either Group 1 or Group 2. Pugh has reported a high proportional limit for crystals of orientation similar to that of specimen M.[4]

Table II lists the proportional-limit stress of each crystal and the resolved shear stress on the slip system indicated by the predominant traces. The average resolved shear stress for the indicated $\{110\}\langle 111\rangle$* slip of Group 1 crystals was 25,500 psi, while that for the indicated $\{112\}\langle 111\rangle$ slip of Group 2 was 13,600 psi. The plane indicated by the slip traces on specimen M seems to correspond to $(\bar{1}01)$. However, the deviation between the observed plane of specimen M and $(\bar{1}01)$ was considerably larger than the deviation found in specimens of Group 1 between the observed plane and $(\bar{1}10)$. The resolved shear stress for

*The appropriate $\langle 111\rangle$ direction was the assumed slip direction for all calculations.

($\bar{1}01$)[111] for specimen M is 55,000 psi, much larger than that for {110}⟨111⟩ slip on Group 1 crystals.

According to classical slip theory, {110}⟨111⟩ and {112}⟨111⟩ would be the two most predominant slip systems in tungsten, since they represent the two most densely populated crystallographic planes and the direction of closest packing. Furthermore, the orientation dependence of the type of slip operative may be calculated if one assumes a particular relation between the critical resolved shear stresses necessary to cause each system to operate. If the ratio $R = \sigma\{110\}\langle 111\rangle/\sigma\{112\}\langle 111\rangle = 1$, that is, if the critical resolved shear stresses for the two types of slip are equal, the slip-system boundaries shown in Fig. 5 will be obtained. If R is less than 1, the two boundaries in the standard stereographic triangle move apart so that {110}⟨111⟩ occurs over a wider range of orientations. If R is greater than 1, the boundaries move together with {112}⟨111⟩ slip occurring for a wider range of orientations. The experimental results based on proportional-limit stresses, given in Table II yield a value of R = 25,500/13,600 = 1.88. For this value of R the boundaries move together such that the ($\bar{1}01$)[111] field is completely eliminated; that is, the experimentally observed shear-stress values predict that there should be no {110}⟨111⟩ slip. This is the essence of the problem to be discussed: the contradiction between the stress data, which predict no {110}⟨111⟩ slip, and the observed distinct slip traces, which indicate a ($\bar{1}10$) plane. It must be realized, however, that there is no logical motion of the boundaries in the stereographic triangle that will explain slip on the planes indicated by the slip traces, since the necessary boundary motion to place Group 1 crystals in the ($\bar{1}01$)[111] field would also put Group 2 crystals, with ($1\bar{1}2$) indicated slip plane, in that field. Another apparent conflict results from the fact that the {110}⟨111⟩ slip system of highest resolved shear stress is ($\bar{1}01$)[111] for all orientations. Thus, if Group 1 crystals do slip on a {110} plane, this plane should be ($\bar{1}01$) not the ($\bar{1}10$) plane indicated by the slip traces.

DISCUSSION

The conflict between these experimental results and the results predicted by previously proposed explanations is great. With the assumption that tungsten must obey the critical resolved shear-stress criterion for the initiation of slip, these results suggest either that gross experimental errors have masked the true behavior or that the true deformation behavior is not obtainable by the conventional techniques used. Both of these possibilities are considered.

With regard to gross experimental errors, the obvious suspect areas are specimen geometry, validity of stress measurements, and validity of slip-plane and slip-direction indexing.

1) Specimen Geometry. The use of longitudinal flats introduces a nonsymmetrical cross section that may lead to bending moments and consequently an erroneous stress measurement. Any bending moments introduced by the small flats used in this investigation have proven to have a negligible effect, since specimens of similar orientations were tested with and without flats (circular cross section) and gave good agreement of proportional-limit stresses.

One might argue that a small bending moment might induce the observed duplex slip. This is not believed to be the case since in Ref. 6 specimens of circular cross section were tested at room temperature and at least six of the nine specimens tested showed duplex slip.

2) Stress Measurements. Using the proportional limit as a measure of the stress for initiation of slip in the absence of a true yield drop probably introduced some error. The proximity of the proportional limit to the onset of any permanent strain, however, was demonstrated during cyclic loading of specimens. The only possibility that would rule out the proportional-limit data would be that slip began at a stress value far below the onset of any measurable permanent strain. This may be a distinct pospossibility, particularly in light of the very low stresses necessary to initiate dislocation motion in some ionic crystals.[10,11] It must be pointed out, however, that other metals obey a critical shear-stress rule based on resolving a stress at or near the proportional limit, for example, zinc,[12] aluminum,[13] and α brass.[14]

3) Slip Directions. Although there are many references in the literature to the stability of the ⟨111⟩ slip direction in bcc metals, it was nevertheless an assumption for the calculations of resolved shear stresses, and an actual determination of the slip direction would seem necessary. Straightforward means of slip-direction determinations were not possible for room-temperature deformation, since deformation was not accompanied by a sufficient rotation of the lattice to allow a determination of the slip direction, as has been done for several fcc metals.[15]

Lattice distortion during deformation results in asterism of the Laue spots on X-ray patterns taken after deformation. It is possible to determine the slip direction by a geometric analysis of the asterism.[16] In the present work, however, the asterism that resulted after room-temperature deformation was so poorly defined that the analysis gave no consistent results.

Several specimens were deformed at 2500°F in the hope that more lattice rotation and distortion would occur at this temperature, allowing slip-direction determination. The axes of these specimens clearly rotated toward the [111] direction. Analysis of asterism also indicated the [111] slip direction. Although these determinations were at 2500°F, they do offer experimental observation of the ⟨111⟩ slip direction in single-crystal tungsten.

4) Slip Plane. As pointed out previously, the possibility exists that dislocation motion was initiated at a stress considerably lower than the observed proportional limit. Accompanying this is the possibility that the active slip plane at such a low stress is not the same as the slip plane indicated from visible traces existing after considerable strain. Also, even though the slip traces of Group 2 crystals were analyzed independently with identical results by two investigators, the traces were faint and wavy, and the analysis may have been in error.

With these possibilities in mind, the proportional-limit data were analyzed with the assumption that the slip-trace data were incorrect. Many "logical"* slip systems and combinations of slip systems were assumed, none of which gave values consistent with a critical resolved shear-stress criterion for slip initiation.

*For example, {112} traces were assumed to be {123}, {122}, and {113}; {112} traces were assumed to be short jogs of {110} slip; {110} traces were assumed to be short jogs of {112} slip.

In addition to these stress calculations, visible slip traces were observed with an electron microscope using a replica technique with magnifications up to 16,000. No evidence was found of short jogs in the slip traces, as might be expected if a given trace is the result of duplex slip.

No evidence was found that would suggest the slip plane to be the plane of maximum shear stress, as found by Leber and Pugh[7] at very high temperatures.

Thus we have considered the slip-trace data to be correct independent of the proportional-limit stress data and found the observed traces to be inconsistent with proposed explanations of slip. We also considered the proportional-limit stress data to be correct independent of the trace data and found that the stress data were also inconsistent with present explanations. The possibility remains that, for any specimen, both the trace data and the stress data were "incorrect", *i.e.*, neither the traces seen nor the proportional-limit stresses measured corresponded to slip initiation. This possibility is discussed below. The ability of the techniques used to reveal the true slip behavior is brought into question when it is remembered that large strains of the order of 10 pct were required to produce visible slip traces. This is an unfortunate, however real, situation. Specimens were examined after small strain increments by optical means, etch-pit techniques, and replica electron microscopy. None of these methods revealed any indication of slip at small strains. Whether this lack of visible slip is unique to the samples used here or is a characteristic of tungsten is not clear. Such behavior is indeed unexpected. In previous work with tungsten by other investigators this problem was either not encountered or not mentioned. In Ref. 6, slip traces were observed after 2 pct strain at 77°K but it is not clear if they were observable at lesser strains nor is there any mention of the amount of strain necessary to produce slip traces at room temperature.

It is then quite obvious that in this investigation appreciable slip occurred before any available method could detect it. This fact coupled with the dependency of strain-hardening rate on orientation shown in Fig. 4 presents a plausible reason for the conflict between measured resolved shear stresses and observed slip systems. The slip system observed at large strains may be a secondary system activated as a result of the primary (first acting) system undergoing strain hardening. This implies that the primary system hardens rapidly and becomes inoperative before it can be detected. If this is the case, resolving the proportional-limit stress on logical slip systems (other than that observed after large strains) would be expected to result in a consistency of resolved shear stresses when the correct primary system is picked. As shown before in the discussion regarding the validity of the slip-plane indices, no consistency of the resolved shear stresses was found. This leads to the conclusion that the primary system was activated at some applied stress below the proportional limit.

Combining these observations makes it appear that the primary system is activated at an applied stress below the proportional limit and strain-hardens rapidly, becoming inoperative before detection. A secondary system is then forced into operation and deformation continues on this secondary system until it produces visible traces at large strains.

CONCLUDING REMARKS

It is believed that this study has brought to light certain difficulties involved in applying conventional research techniques to the study of the deformation of tungsten at room temperature. It has been shown that tungsten single crystals undergo considerable plastic deformation before any visible evidence of slip is revealed and determinations of slip systems and resolved shear stresses based upon the visible slip traces result in values inconsistent with the critical resolved shear-stress criterion. In light of the observations of this investigation the following points are suggested.

1) The deformation behavior of single-crystal tungsten is highly orientation-dependent, the proportional-limit stress, yield behavior, and strain-hardening rate changing appreciably with orientation.

2) The primary slip system in tungsten becomes activated at applied stresses below the conventionally measured proportional-limit stress.

3) The primary system becomes inoperative due to rapid strain hardening and therefore remains unidentifiable by conventional techniques.

4) When slip traces are visible, these probably represent a secondary system which has become operative due to strain hardening of the primary system.

REFERENCES

[1]J. H. Hoke and R. Maddin: *J. Mech. Phys. Solids*, November, 1956, vol. 5, no. 1, pp. 26-40.

[2]N. K. Chen and R. Maddin: *AIME Trans.*, 1951, vol. 191, pp. 937-44.

[3]F. S. Goucher: *Phil. Mag.*, 1924, vol. 48, 6th ser., pp. 800-19.

[4]J. W. Pugh: On the Purification of Tungsten by Electron Beam Refining, Proceedings of First Symposium on Electron-Beam Melting, pp. 89-93, Alloyd Research Corp., Watertown, Mass., 1959.

[5]Russell H. Atkinson and Staff of Metals Research Group: Physical Metallurgy of Tungsten and Tungsten Base Alloys, p. 145, WADD TR 60-37, Westinghouse Electric Corp., May, 1960.

[6]Harvey W. Schadler: *Trans. Met. Soc. AIME*, August, 1960, vol. 218, pp. 649-55.

[7]Sam Leber and J. W. Pugh: *Trans. Met. Soc. AIME*, October, 1960, vol. 218, pp. 791-93.

[8]W. R. Witzke: Zone Melting of Tungsten by Electron Bombardment, Proceedings of First Symposium on Electron-Beam Melting, pp. 73-81, Alloyd Research Corp., Watertown, Mass., 1959.

[9]Charles S. Barrett: *Structure of Metals*, 2nd ed., p. 40, McGraw-Hill Book Co., New York, 1952.

[10]Carl A. Stearns, Ann E. Pack, and Robert A. Lad: Ductile Ceramics, I – Factors Affecting the Plasticity of Sodium Chloride, Lithium Fluoride, and Magnesium Oxide Single Crystals, NASA TN D-75, NASA, Washington 25, D. C., 1959.

[11]R. J. Stokes, T. L. Johnston, and C. H. Li: *Trans. Met. Soc. AIME*, June, 1959, vol. 215, pp. 437-44.

[12]S. Harper and A. H. Cottrell: *Proc. Phys. Soc. (London)*, 1950, vol. 63, no. 365B, pp. 331-38.

[13]F. D. Rosi and C. H. Mathewson: *AIME Trans.*, September, 1950, vol. 188, pp. 1159-67.

[14]Raymond W. Fenn, Jr., Walter R. Hibbard, Jr., and Henry A. Lepper, Jr.: *AIME Trans.*, January, 1950, vol. 188, pp. 175-81.

[15]Constance F. Elam: *Distortion of Metal Crystals*, pp. 19-21, Clarendon Press, Oxford, 1935.

[16]E. J. Rapperport and C. S. Hartley: *Trans. Met. Soc. AIME*, October, 1960, vol. 218, pp. 869-76.